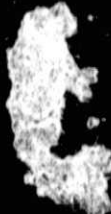


General Disclaimer

One or more of the Following Statements may affect this Document

- This document has been reproduced from the best copy furnished by the organizational source. It is being released in the interest of making available as much information as possible.
- This document may contain data, which exceeds the sheet parameters. It was furnished in this condition by the organizational source and is the best copy available.
- This document may contain tone-on-tone or color graphs, charts and/or pictures, which have been reproduced in black and white.
- This document is paginated as submitted by the original source.
- Portions of this document are not fully legible due to the historical nature of some of the material. However, it is the best reproduction available from the original submission.



SILICON INGOT CASTING - HEAT EXCHANGER METHOD MULTI-WIRE SLICING - FIXED ABRASIVE SLICING TECHNIQUE

Phase III

(NASA-CR-158734) SILICON INGOT CASTING,
HEAT EXCHANGER METHOD MULTI-WIRE SLICING,
FIXED ABRASIVE SLICING TECHNIQUE. PHASE 3:
SILICON SHEET GROWTH DEVELOPMENT FOR THE
LARGE AREA SHEET (Crystal Systems, Inc.,

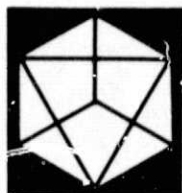
N79-26981

HC A03 / MF A01
Unclas
G3/76 27825



**QUARTERLY PROGRESS REPORT NO. 2
BY
FREDERICK SCHMID AND CHANDRA P. KHATTAK**

Covering Period from January 1 through March 31, 1979
Report Issued: April 1979
JPL Contract No. 95-373



CRYSTAL SYSTEMS INC.

Snetland Industrial Park, 35 Congress Street, Salem, Mass. 01970

SILICON INGOT CASTING - HEAT EXCHANGER METHOD
MULTI-WIRE SLICING - FIXED ABRASIVE SLICING TECHNIQUE

(PHASE III)

Silicon Sheet Growth Development for the
Large Area Sheet Task of the
Low-Cost Solar Array Project

Quarterly Progress Report No. 2

by

Frederick Schmid and Chandra P. Khattak

Covering Period from January 1 through March 31, 1979

Report Issued: April 1979

JPL Contract No. 954373

CRYSTAL SYSTEMS, INC.

35 Congress Street
Salem, MA 01970

The JPL Low-Cost Solar Array Project is sponsored by the U. S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology by agreement between NASA and DOE.

This report contains information prepared by Crystal Systems, Inc., under JPL subcontract. Its content is not necessarily endorsed by the Jet Propulsion Laboratory, California Institute of Technology, National Aeronautics and Space Administration or the U. S. Department of Energy.

TABLE OF CONTENTS

	Page
TABLE OF CONTENTS.	iii
ACKNOWLEDGMENT	iv
ABSTRACT	v
SILICON INGOT CASTING--HEAT EXCHANGER METHOD . .	1
Crystal Growth	1
Crucible Development	8
Purification of Heat Zone	11
Design of Furnace Chamber	19
MULTI-WIRE SLICING--FIXED ABRASIVE SLICING TECHNIQUE.	20
Testing.	20
Machine Development.	21
Blade Development.	22
CONCLUSIONS	33
REFERENCES	35
MILESTONES	36

ACKNOWLEDGEMENT

The authors wish to acknowledge the valuable assistance and contributions from John A. Lesiczka, Barbara B. Nelson, and Maynard B. Smith.

ABSTRACT

Several 20 cm diameter silicon ingots, up to 6.3 kg, have been cast with good crystallinity.

It has been found that the graphite heat zone can be purified by heating it to high temperatures in vacuum. This is important in reducing costs and purification of large parts.

Electroplated wires with 45 μm synthetic diamonds and 30 μm natural diamonds have shown good cutting efficiency and lifetime. During slicing of a 10 cm x 10 cm workpiece, jerky motion occurred in the feed and rocking mechanisms. This problem is being corrected and at the same time modifications are being made to reduce the weight of the bladehead by 50%.

SILICON INGOT CASTING -- HEAT EXCHANGER METHOD

Solar cells fabricated from HEM-cast silicon ingots have shown 15% conversion efficiency. This has been achieved without optimization of material or cell processing parameters. In addition, HEM is the only process which yields single-crystal, square silicon ingots. All the essential features of the process have been demonstrated and the present emphasis is on scale-up.

Crystal Growth

It is desirable to cast square cross-section ingots in order to increase the solar cell packing efficiency in arrays and thereby achieve cost reductions. Ingots of 10 cm x 10 cm cross-section have been cast and it is intended to scale up the process to 15 cm x 15 cm size followed by 20 cm x 20 cm. The intermediate size of 15 cm x 15 cm is being attempted for two reasons. Firstly, it is a sequential scale up and crystal growth problems, if any, encountered can be addressed easily. Secondly, the relationship between heat zone and crucible size can be established.

Custom-order square crucibles have long delivery periods. Therefore, 20 cm diameter crucibles have been purchased. These

crucibles are intended to study the crystal growth of larger size silicon ingots. The furnace chamber has been rebuilt with graphite parts, heat conducting pipes, etc., for operation for 20 cm diameter ingots.

Runs 301 and 302 (details in Table I) were carried out using 20 cm diameter crucibles. The charge melt-down cycle progressed smoothly; however, during the "seeding" operation the silicon melt flowed out of the crucible. It was felt that on a large diameter, such as a 20 cm diameter crucible, because of plastic flow range of silica and differential thermal expansion coefficients of silica and graphite, the silica crucible-graphite plug-silicon seed joint was deformed and resulted in spillage of the melt. The deformation of silica crucible at high temperatures has been observed in 15 cm diameter crucibles;¹ this has resulted in breakdown of crystallinity of the ingot. It was felt that in larger sizes the deformation is correspondingly more. Therefore, the crucible has to be supported so that crucible deformation does not result in loss of molten silicon. A graphite support plate was designed on which the deformation of the crucible at high temperatures and weight of the melt will not result in stresses on the crucible-plug-seed interface.

In preparation for casting 20 cm diameter ingots it had been established that a low-cost grade of graphite be used which does not deform substantially on contact with molten silicon. The grade of graphite arrived at after evaluation was ATJ.²

TABLE I. TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

RUN	PURPOSE	SEEDING		GROWTH CYCLE			REMARKS
		FURN. TEMP. ABOVE M.P. °C	H.E. TEMP. BELOW M.P. °C	RATE OF DECREASE H.E. TEMP. °C/HR.	FURN. TEMP. °C	GROWTH TIME IN HOURS	
301-C	Cast 20 cm diameter ingots	-	-	-	-	-	Run aborted due to distortion of crucible.
302-C	Cast 20 cm diameter ingots	-	-	-	-	-	Run aborted due to distortion of crucible.
303-C	Cast 20 cm diameter ingots	13	341	130	13	4.5	3.8 kg ingot cast.
304-C	Cast 20 cm diameter, 5.3 kg ⊕ ingot	15	277	185	15	6.5	Some attachment of crucible.
305-C	Cast 20 cm diameter, 4.7 kg ingot	11	280	132	11	6.5	Very limited attachment. Good crystallinity.
306-C	Crucible develop- ment with 10 cm x 10 cm ingot	19	236	115	19	5.5	Very limited attachment. Very good crystallinity.
307-C	Crucible develop- ment with 10 cm x 10 cm ingot	3	223	170	3	7.5	Power failure at end of growth cycle. Good de- lamination of crucible and very good crystallinity.
308-C	Crucible develop- ment with 15 cm diameter ingot	<3	260	125	1	6.5	Attachment of crucible caused cracking.

. . . cont.

TABLE I. TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES (Cont.)

RUN	PURPOSE	SEEDING		GROWTH CYCLE		GROWTH TIME IN HOURS	REMARKS
		FURN. TEMP. ABOVE M.P. °C	H.E. TEMP. BELOW M.P. °C	H.E. TEMP. °C/HR.	FURN. TEMP. °C		
309-C	Crucible develop- ment with 15 cm diameter ingot	<3	222	160	1	7.0	Attachment of crucible in localized area.
310-C	Crucible develop- ment with 15 cm diameter ingot	-	-	-	-	-	Run terminated as crucible cracked
311-C (C)	Crucible develop- ment with 15 cm diameter ingot	5	254	132	5	6.8	Minimal attachment of crucible in localized areas.
312-C	Crucible develop- ment with 15 cm diameter ingot	13	252	122	13	8.6	Crucible attachment to ingot
313-C	Cast 20 cm diam- eter, 5.3 kg ingot	10	224	120	10	7.0	Crucible attachment in some areas.
314-C	Cast 20 cm diam- eter, 5.3 kg ingot	36	172	147	36	9.0	Crucible attachment in localized areas. Good crystallinity.
315-C	Cast 20 cm diam- eter, 6.3 kg ingot	4	168	154	4	7.0	Minimized crucible attach- ment in localized areas.
316-C	Crucible develop- ment with 15 cm diameter ingot	5	165	158	5	8.3	Attachment of crucible to ingot.

During the spill of molten silicon in runs 301 and 302 it was found that even though molten silicon flowed around the ATJ graphite plug it maintained its dimensional shape, further emphasizing the suitability of this grade. Another feature incorporated in the heat zone of the furnace was the modification of the hearth plate. With this modification the damage to the furnace parts should be minimized if liquid silicon spills as occurred in runs 301 and 302.

Run 303 was carried out using a modified support plate for the crucible. A 3.8 kg, 20 cm diameter ingot was cast. The as-cast surface of the ingot is shown in Figure 1. The adherent pieces of silica are visible on the surface. Delamination of the crucible was very good except in a few localized spots. It is felt that automation in the heat treatment of crucibles is necessary to uniformly grade the structure of the crucible. A view of the top surface of the ingot is shown in Figure 2. It can be seen that slight cracking was obtained on the surface of the ingot where the crucible was not properly graded.

In runs 304-C, 313-C and 314-C, 20 cm diameter, 5.3 kg ingots were cast. Some attachment of the crucible to the ingot caused cracking of the boule. In run 305-C a 20 cm diameter, 4.7 kg ingot was cast. The attachment of the crucible was limited to a few small areas which caused some "chipping" but the ingot was essentially crackfree (Figure 3). Figure 4 shows the bottom of the boule with the impression of a 2.5 cm diameter

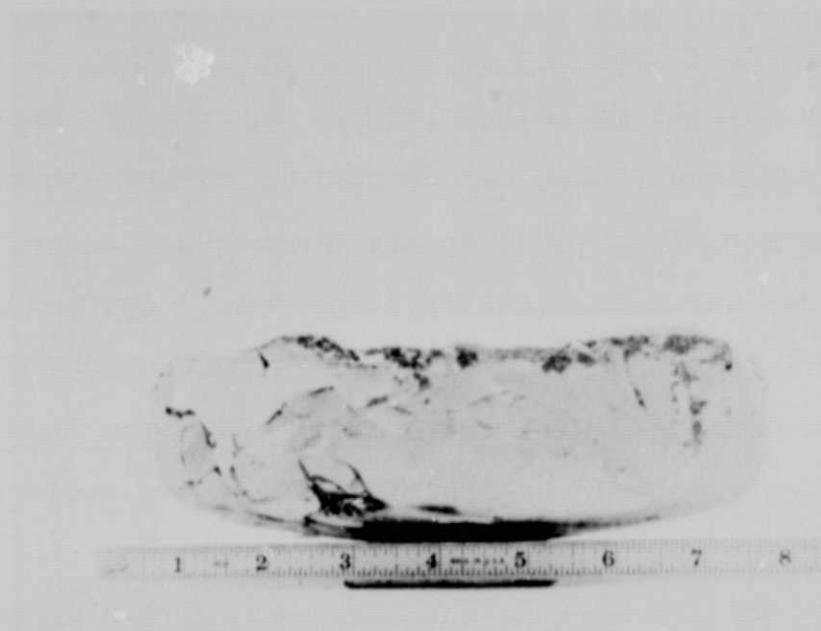


Figure 1. View of the as-cast surface of a 3.8 kg, 20 cm diameter ingot cast in run 303-C.



Figure 2. View of the top surface of ingot in Figure 1.

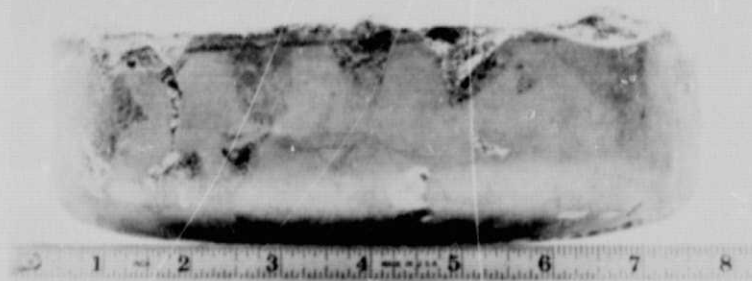


Figure 3. A 4.7 kg silicon ingot cast in run 305-C.

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

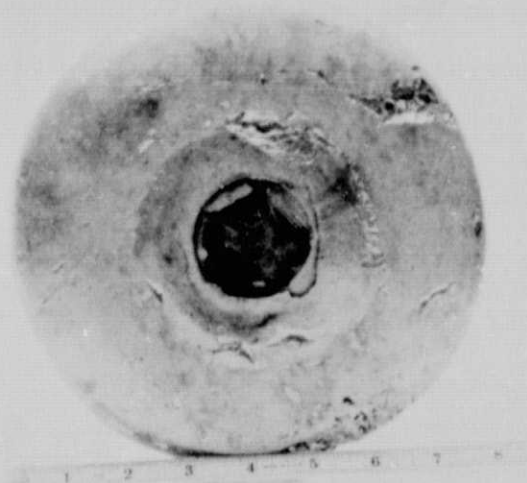


Figure 4. A view of the bottom of the ingot shown in Figure 1.

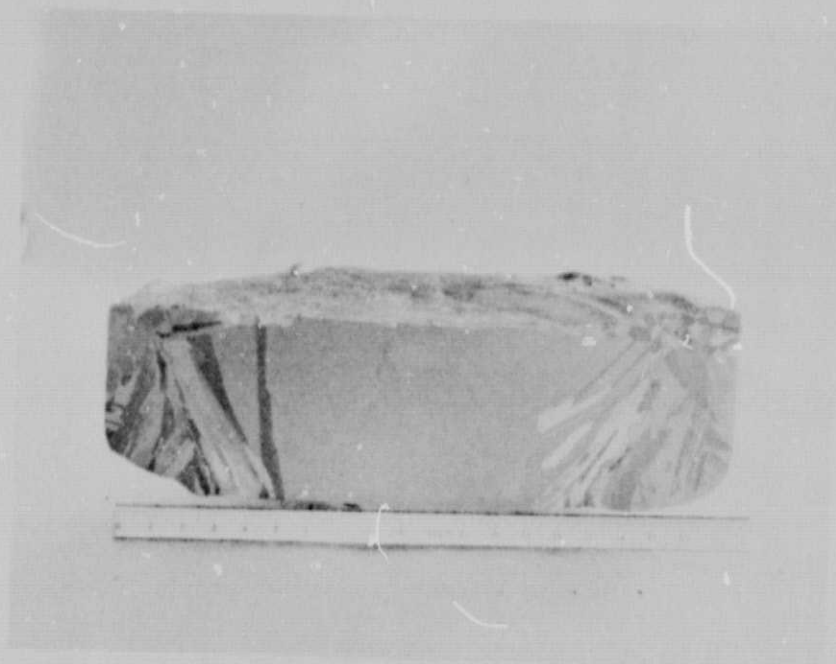
heat exchanger. A polished and etched section of boule 305-C and 314-C is shown in Figure 5. The central area is single crystal with some twinning and large grains on the sides. Careful examination of the bottom of the ingots shows that the crucible had sagged at high temperature in the area where there was breakdown in crystallinity. The consequence of distortion of the crucible resulted in crystal growth being initiated off the crucible rather than off the growing solid-liquid interface.

In run 315-C the 20 cm diameter ingot was scaled up to 6.3 kg. Very minimal attachment of the crucible in localized areas was observed. Figure 6 shows a view of the cast ingot.

Crucible Development

It has been reconfirmed that a graded structure² in the silica crucible is necessary to prevent ingot cracking. The structure has to be uniform also so that localized attachment of crucible to the ingot is avoided. In large size crucibles, such as 20 cm diameter, it is difficult to achieve a uniform graded structure. At the present time the crucible is heat treated manually with a flame torch. Under such circumstances it is difficult to achieve uniformity of structure over the entire surface of the crucible.

In run 306-C and 307-C 10 cm x 10 cm square cross-section crucibles were studied for optimum structure to cause delamination of crucible. Such a small size is especially difficult to grade uniformly since it does not allow good maneuverability of the



REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

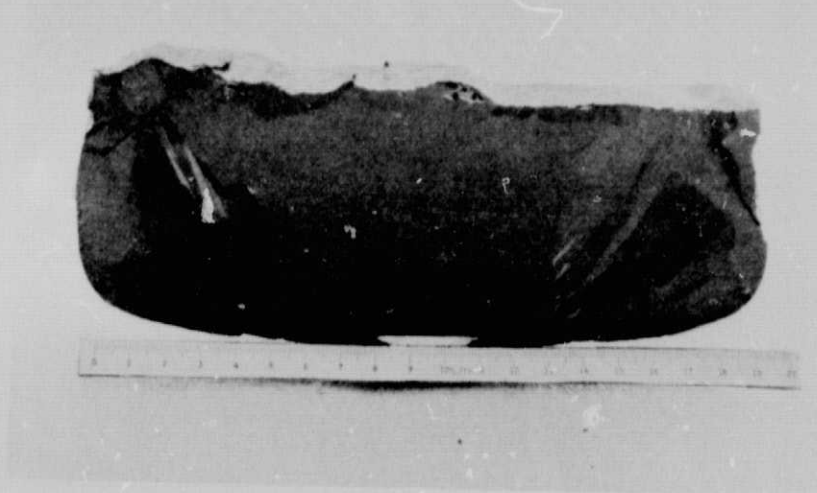


Figure 5. A polished and etched section of ingot cast
in run 305-C (top) and 314-C (bottom).



Figure 6. View of ingot cast in run 315-C.

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

torch within the crucible. Except for localized areas very good delamination of the crucible was achieved.

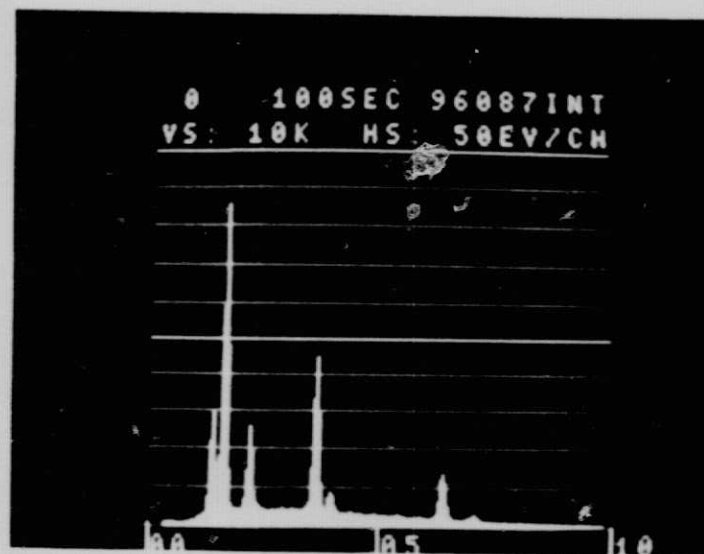
For a production process it will be necessary to set up an automated method, such as induction heating, to develop a graded structure. This will allow heat treatment of large crucibles with good uniformity.

Purification of Heat Zone

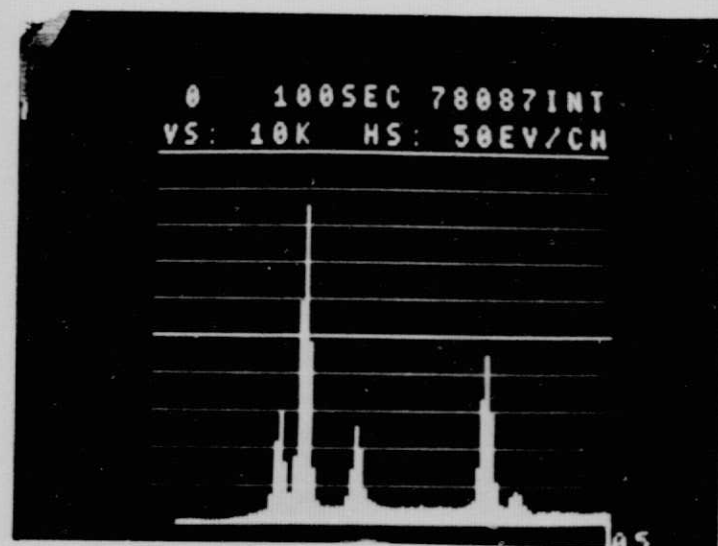
One of the essential requirements of silicon processing is a purified heat zone. The graphite available contains many impurities which contaminate the furnace and the silicon. Analysis of metallic elements in a graphite piece are shown in Table II. It can be seen that most of the contaminants are high vapor pressure elements. In industry the graphite parts that are used in the furnace are purified by a halogen treatment. This process adds substantially to the cost of the heat zone. Besides the cost, purification is a problem when large sizes are involved. It is intended to use the HEM to grow 30 cm x 30 cm x 30 cm silicon ingots and, therefore, very large size graphite parts by purifying as a heat zone. The furnace was heated in 0.1 torr vacuum to about 1600°C, above the temperature it is normally used, soaked for about 12 hours and then the power was shut off. On opening the furnace a smell similar to that of a sulphur compound was evident and a deposit was found on the cooler sections of the furnace. This deposit was examined by EDAX for qualitative

analysis. Three separate examinations were made using electron beam accelerating voltages of 10 and 20 KV and low and high area magnifications. Primary concern was to be sure that the indications of aluminum presence were from the sample and not from the aluminum stud the sample was mounted on. The EDAX spectra are given in Figure 7-9 for the different operating conditions. Consistent indications of Cr, Al, Si, S, Ca and Fe were found with the possible presence of W and P. A comparison of the elements found in the deposit with the data in Table II shows that most of the high level impurities in the graphite were accounted for in the deposit.

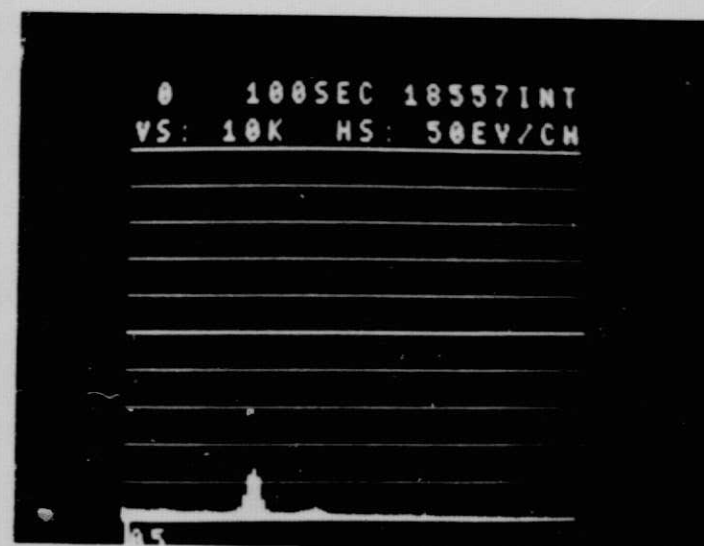
Run 306-C was carried out after the purification run. In this run a 1.9-2.1 Ω -cm resistivity seed was used and the melt was doped to bring the resistivity to about 0.5 Ω -cm. The cast ingot had very good crystallinity and four-probe resistivity measurements were carried out in a grid pattern as shown in Figure 10. The data is shown in Table III. It can be seen rather uniform resistivity close to the target value was achieved in the sample. The last material to freeze showed very low resistivity. This confirms that (i) the heat zone can be purified by heating to high temperatures in vacuum (ii) rather uniform resistivity is achieved in HEM cast ingots and (iii) impurities are rejected in the last material to freeze.



REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

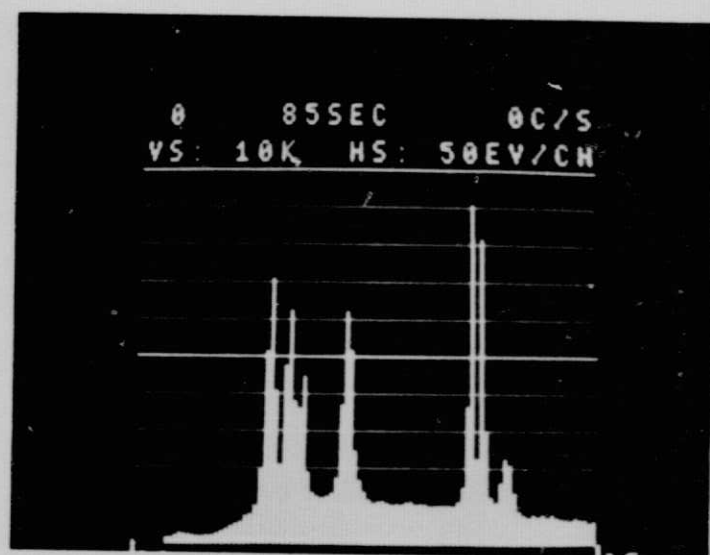
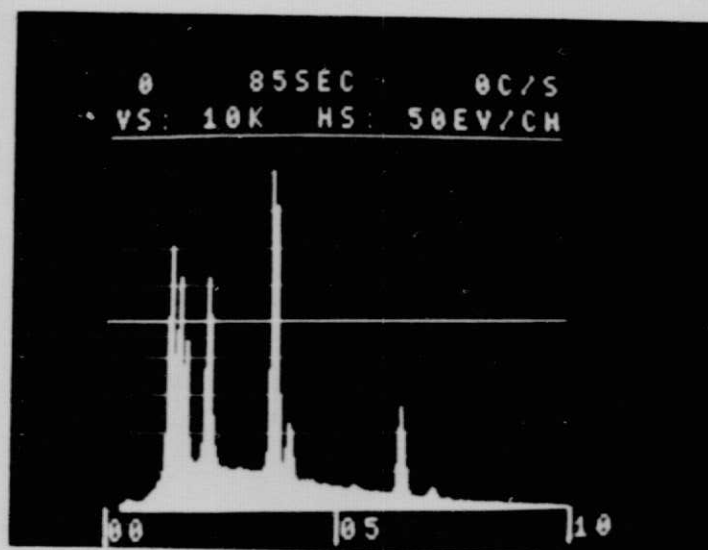


Cr(Lα) Al(Kα) Si(Kα) S(K) Ca(K) Cr(Kα)
[W(M)?] [P(M)?]



Cr(Kβ) Fe(Kα) Fe(Kβ)

Figure 7. EDAX, 20KV, X100.



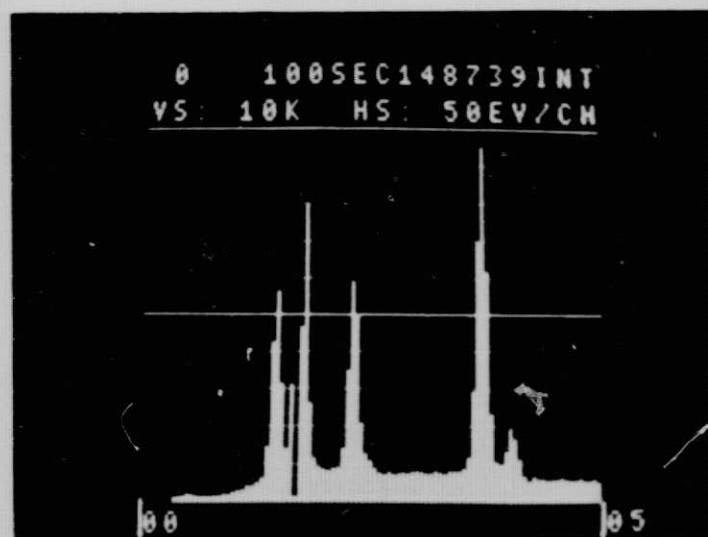
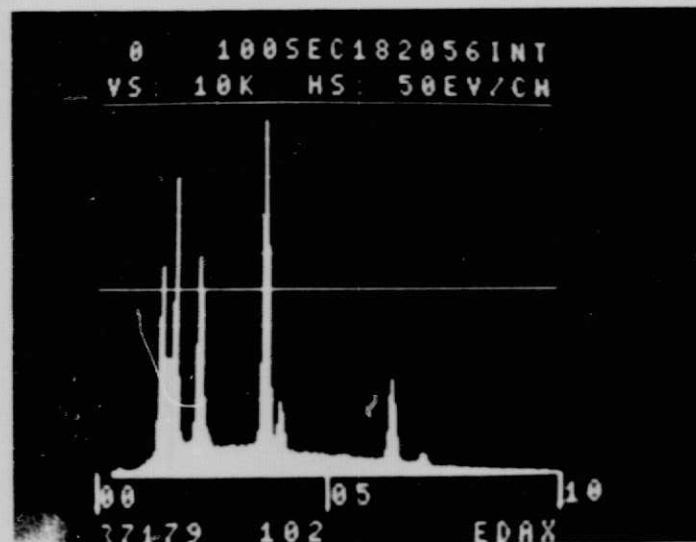
Cr(Lα) Al(Kα) Si(Kα) S(K) Ca(K) Cr(Kα)
[W(M)?] [P(M)?]



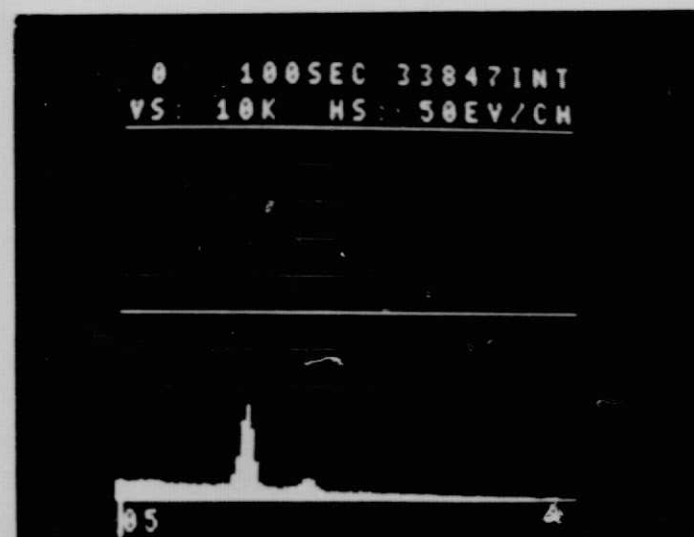
Cr(Kβ) Fe(Kα) Fe(Kβ)

Figure 8. EDAX, 10KV, X32,000.

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR



Cr(Lα) Al(Kα) Si(Kα) S(K) Ca(K) Cr(Kα)
[W(M)?] [P(M)?]



Cr(Kβ) Fe(Kα) Fe(Kβ)

Figure 9. EDAX, 10KV, X100.

TABLE II. TYPICAL IMPURITIES OF GRAPHITE USED
IN HEM FURNACE MEASURED BY
SPECTROGRAPHIC ANALYSIS

Element	Content (ppm)
Mg	13.0
Si	> 100.0
Fe	120.0
Ni	1.0
Al	100.0
Cu	12.0
V	10.0
Ti	23.0
Ca	325.0
Pb	9.0
Cr	5.0
Mo	N.D.*
Mn	10.0
B	N.D.
TOTAL	> 728.0

* Not detected.

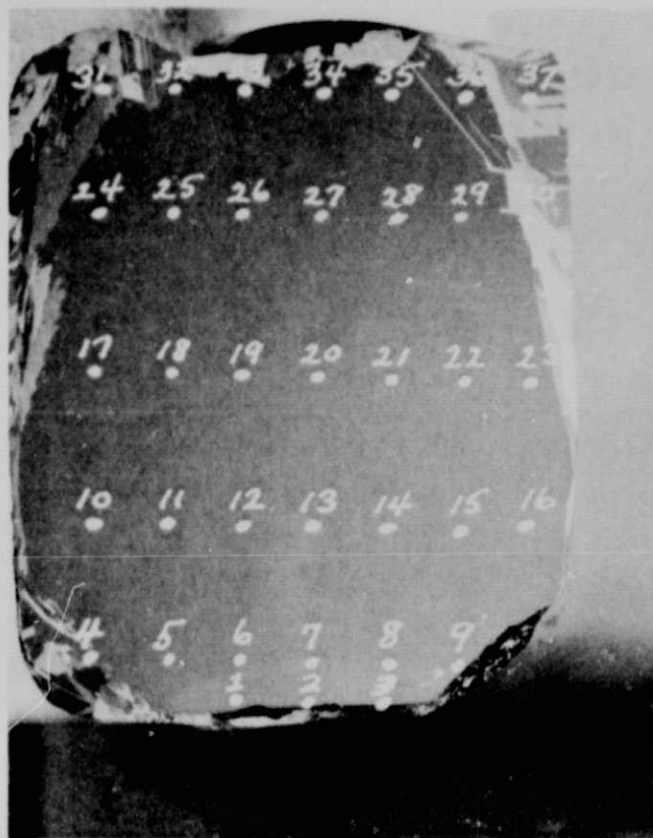


Figure 10. A polished and etched section of a 10 cm x 10 cm square ingot cast in run 306-C along with resistivity grid pattern

TABLE III. RESISTIVITY PROFILE FOR RUN 306
(Numbers Correspond to Figure 10)

0.06 [*] (#31)	0.12 (#32)	0.06 (#33)	0.06 (#34)	0.13 (#35)	0.12 (#36)	(#37)
0.19 (#24)	0.21 (#25)	0.21 (#26)	0.22 (#27)	0.21 (#28)	0.22 (#29)	0.23 (#30)
0.23 (#17)	0.24 (#18)	0.24 (#19)	0.32 (#20)	0.26 (#21)	0.33 (#22)	0.32 (#23)
0.32 (#10)	0.30 (#11)	0.28 (#12)	0.33 (#13)	0.28 (#14)	0.33 (#15)	0.30 (#16)
0.32 (#4)	0.35 (#5)	0.39 (#6)	0.34 (#7)	0.38 (#8)	0.32 (#9)	
		1.92 (#1)	2.02 (#2)	1.86 (#3)		

Ω -cm

Design of Furnace Chamber

The maximum size of ingot that can be grown with the existing furnace chamber is slightly larger than 20 cm diameter. The goal of the present program is to cast 20 cm x 20 cm square cross-section ingots. A new larger furnace chamber has been designed which will allow ingot sizes up to 30 cm x 30 cm size. This design is now completed.

MULTI-WIRE SLICING -- FIXED ABRASIVE SLICING TECHNIQUE

The material utilization feature of Fixed Abrasive Slicing technique (FAST) has been demonstrated on 4 cm x 4 cm workpiece using a modified Varian 686 slicer. A high speed slicer has been used to slice 10 cm diameter silicon ingot at 19 wafers per cm. During the current quarter slicing of 10 cm x 10 cm silicon has been carried out and modifications of the slicer are in progress to slice 25 wafers per cm. Blade development has continued on the modified 686 machine.

Testing

A 10 cm x 10 cm square cross-section silicon workpiece was sliced in run 301-SX. The silicon ingot was cut to size from a 15 cm diameter ingot cast by the Heat Exchanger Method. The top of the ingot coincided with the as-cast surface of the ingot. The wires used were commercially impregnated 45 μ m natural diamonds with a 7.5 μ m electroless nickel plating. Feed forces were 41.3 gms per blade and the average cutting rates over the entire cross-section were 0.060 mm/min. The cutting rates of runs 301-S and 2-002-S were similar; however, the area of sliced wafers was 100 cm² and 78.5 cm²

respectively. It was found that as the cutting progressed the vibrations increased and the rocking of the workpiece had to be limited in order to decrease the vibrations. These vibrations were due to a jerky rocking motion at the end of each stroke. In addition, the feed mechanism produced non-uniform feed forces due to a hysteresis in the cylinder that actuated the feed mechanism. This is being corrected along with other modifications to the slicer.

Machine Development

The slicing experiments with the high speed slicer have been at 19 wafers per cm. Even though good vibration isolation has been achieved the workpiece suffered from jerky rocking mechanism and from a hysteresis in the feed mechanism. It is intended to slice 25 wafers per cm and these problems will limit the slicing performance. Moreover, it is desirable to achieve higher surface speeds for efficient slicing with fixed diamond abrasive. This can be achieved by lightening the bladehead. The present bladehead is twice as long as that on the modified Varian 686 slicer yet it is half the weight. It is intended to modify the bladehead carriage so that its weight is reduced by another 50%. This is expected to give surface speeds of 500 feet per minute. These modifications have been detailed and fabrication of parts is in progress.

Blade Development

It has been established that better slicing performance is achieved when fixed diamond wire blades are used at higher speeds. Optimum blades developed for the modified 686 slicing machine will perform better with the high speed slicer. In this way direct comparisons can be made with previous results and economies can be exercised by continuing blade development with the modified 686 slicer. Moreover, this work can be continued in parallel with machine modifications of high speed slicer.

During the current time period two types of electroplated wires were tested. Both the blade packs were plated by two new vendors who show good potential for reducing kerf. An unused section of a 45 μm electroplated, steel-core wire is shown in Figure 11. In these wires the kerf was reduced by decreasing the nickel plating during electroplating. During use in runs 301-S and 302-S (details in Table IV) good cutting rates were achieved. However, many wires broke due to corrosion problems. During electroplating, the acids attack the steel core, especially near the clamps of the blade pack. When the wires are stretched for slicing stress-corrosion cracking results in degradation of the wires.

The electroplated wires used in runs 307-S through 311-S were 45 μm synthetic diamonds plated on tungsten core. An unused section of the wire was examined with the SEM (Figure 12). The three views are with the wire rotated 120° to see good

TABLE IV. SILICON SLICING SUMMARY

RUN	PURPOSE	FEED		AVERAGE CUTTING RATE		WIRE TYPE	REMARKS
		FORCE/BLADE lb.	gm	mil/min	mm/min		
301-S	Test electroplated steel core wires	0.077	34.9	2.85	0.072	Steel core electroplated with 45 μ m diamonds	Good cutting rates. Poor yield. Corrosion problems with wire.
302-S	Lifetest	0.078	35.3	2.68	0.068	Same as 301-S	Many wires broken during run due to corrosion.
303-S	Test impregnated wire with 7.5 μ m sheath	0.077	34.9	1.99	0.051	5 mil, 0.125 mm W/core; 0.3 mil, 7.5 μ m copper sheath; 45 μ m diamond CSI impregnation. 0.3 mil, 7.5 μ m nickel plated.	Good wafer quality. 90% yield.
(23) 304-S	Lifetest	0.078	35.3	0.98	0.025	Same as 303-S.	Run discontinued due to poor cutting rates.
305-S	Test impregnated wire with 12.5 μ m sheath	0.077	34.9	2.64	0.067	5 mil, 0.125 mm W/core; 0.5 mil, 12.5 μ m copper sheath; 45 μ m diamond CSI impregnation. 0.3 mil, 7.5 μ m electroless nickel plated.	Good cutting rates and wafer quality. 95% yield.
306-S	Lifetest	0.078	35.4	1.27	0.032	Same as 305-S	Poor cutting rates due to diamond pull-out. 54% yield.
307-S	Test electroplated wires	0.075	34.2	2.40	0.061	W core electroplated with 45 μ m synthetic diamonds	Poor yield due to uneven nickel buildup.
308-S	Lifetest	0.075	34.2	1.60	0.041	Same as 307-S	Stroke shortened to 6" to avoid nickel buildup problem on ends. 45% yield.

TABLE IV. SILICON SLICING SUMMARY (Cont.)

RUN	PURPOSE	FEED		AVERAGE		WIRE TYPE	REMARKS
		FORCE/BLADE lb.	gm	CUTTING RATE mil/min	mm/min		
309-S	Lifetest continuation	0.075	34.2	1.17	0.030	Same as 307-S	Lower cutting rates due to limited stroke length 40% yield.
310-S	Lifetest continuation	0.075	34.2	1.27	0.032	Same as 307-S	Cutting rates constant. 38% yield.
311-S	Lifetest continuation	0.075	34.2	1.40	0.036	Same as 307-S	Cutting rates constant. 28% yield.
312-S	Test electroplated impregnated wires	N/A	N/A	N/A	N/A	Commercially impregnated wire electroplated with nickel	Run aborted. Poor cutting performance.
313-S	Test electroplated impregnated wires	N/A	N/A	N/A	N/A	Same as 312-S	Run aborted. Diamond pull-out from wires.
314-S	Test electroplated wires	0.070	31.9	2.5	0.064	5 mil, 0.125 mm, W core electroplated with 30 μ m diamonds	Very good wafer quality; 100% yield
315-S	Lifetest	0.070	31.9	2.02	0.051	Same as 314-S	Good wafer quality; 98% yield.
316-S	Lifetest continuation	0.070	31.9	1.75	0.044	Same as 314-S	Good wafer quality; 87% yield.
317-S	Lifetest continuation	0.070	31.9	1.89	0.048	Same as 314-S	Good wafer quality; 77% yield.

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR



Figure 11. View of a longitudinal section of electroplated wire. The kerf was reduced by decreasing the nickel plating.

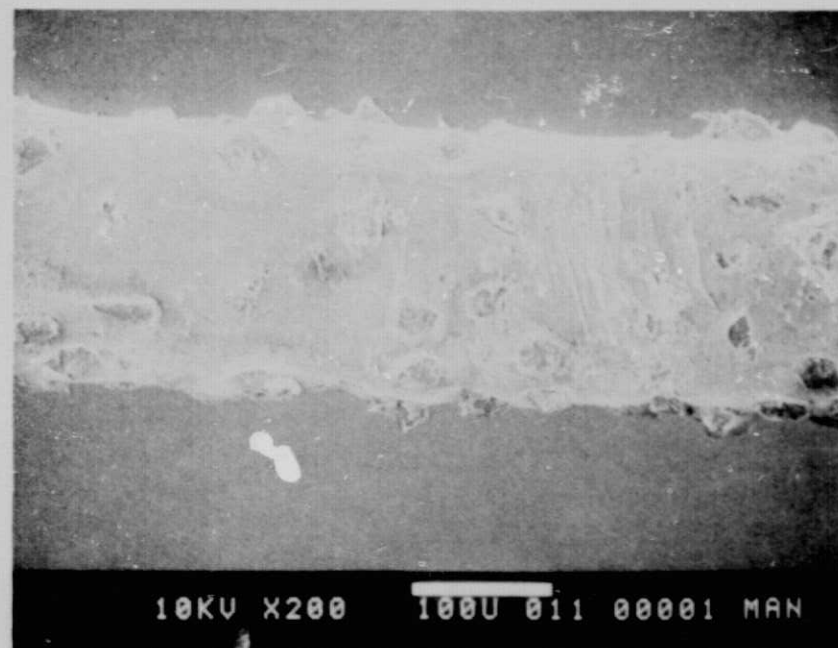
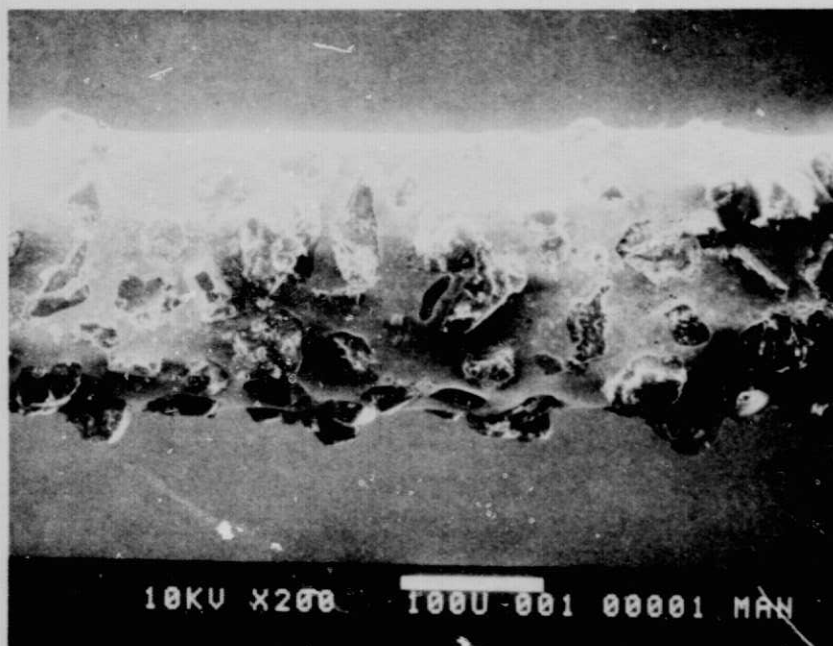


Figure 12. Three views of unused electroplated wire rotated 120° .

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS

uniformity and high concentration of the diamonds. A close-up of a few diamonds (Figure 13) shows that the diamonds are of "blocky" nature but are held well in the plating. Examination of cross-sections of this wire in two different areas (Figure 14) showed that the nickel buildup was uneven. Near the ends of the stroke length (Figure 14a) the nickel buildup was higher and buried the diamonds as compared to the central area of wire (Figure 14b). During testing with these wires the uneven nickel buildup and burying of diamonds near the ends of the stroke length resulted in poor yields of wafers. Testing was continued with a shortened stroke length with a view to study the degradation of nickel/diamond bond and diamond concentration. Examination of the wires after use in run 307-S (Figure 15) and after five runs, 307-S through 311-S, (Figure 16) shows that diamond concentration is still high and there appears to be no diamond pull-out.

The encouraging results achieved in runs 307-S through 311-S pursued the electropolating of a bladepack with 30 μ m natural diamonds. It was expected that the natural diamonds will give better cutting performance and the smaller diamond size will reduce the kerf. These wires were tested in runs 314-S through 317-S. It was found that in the very first test 100% yield was achieved with good cutting rates. During subsequent runs the cutting rates decreased. However, high yields were maintained. Prior to run 317-S the wires were dressed

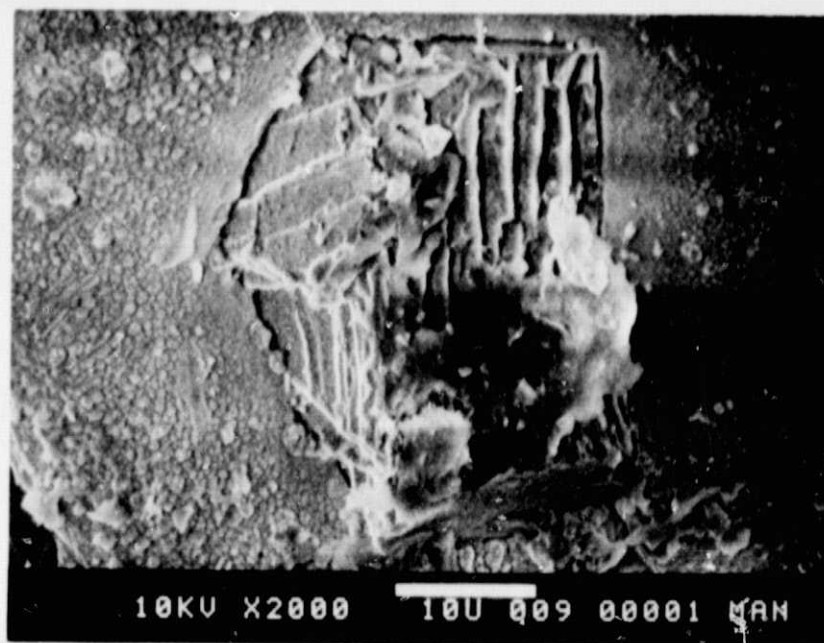
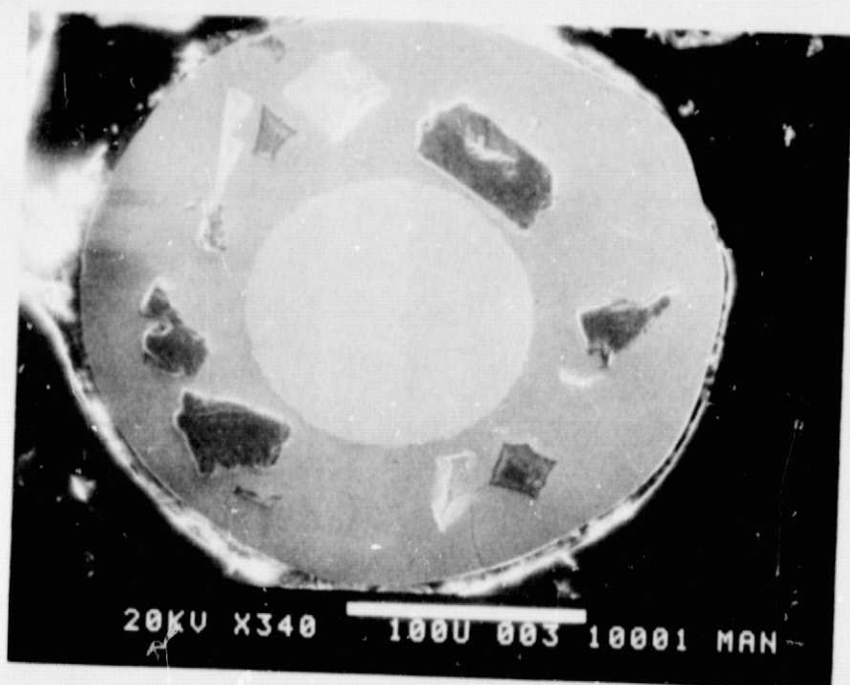
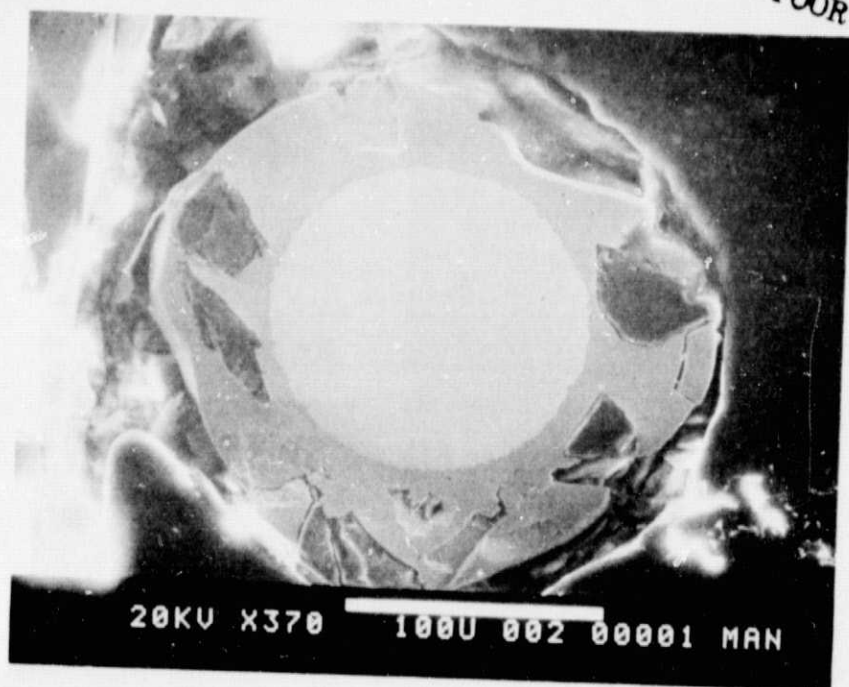


Figure 13. High magnification view of wire in Figure 6 showing "blocky" diamonds.



(a)

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR



(b)

Figure 14. Cross-sectional view of wire in Figure 6 showing (a) high nickel buildup near end and (b) central area.

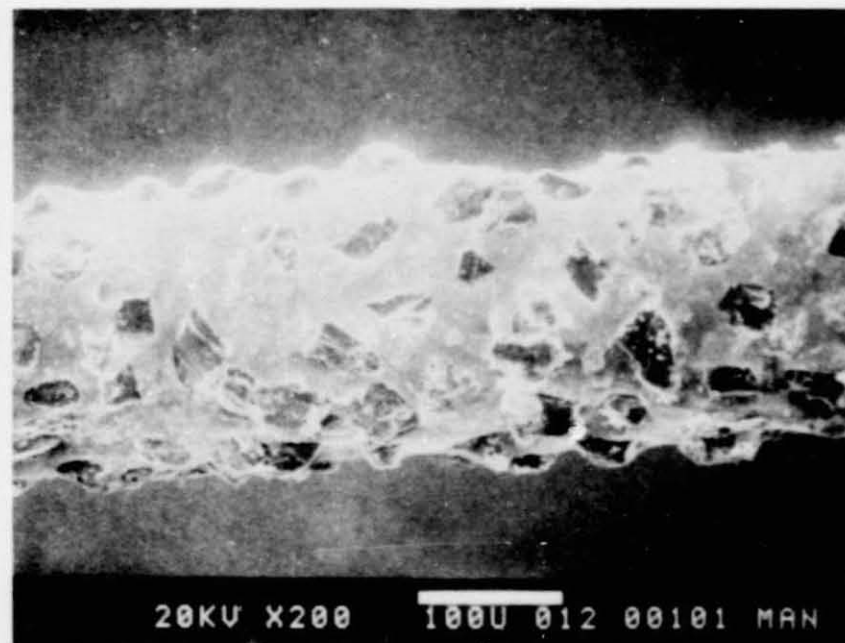
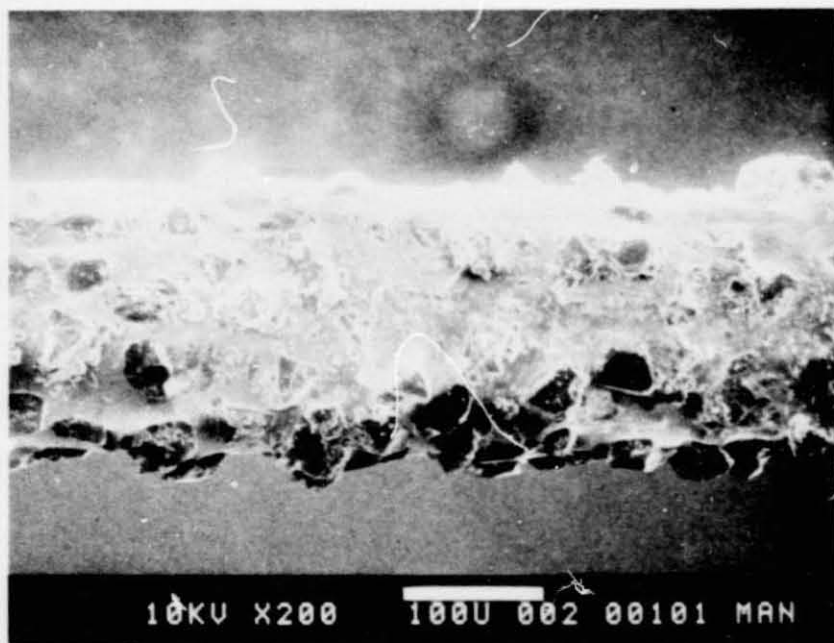
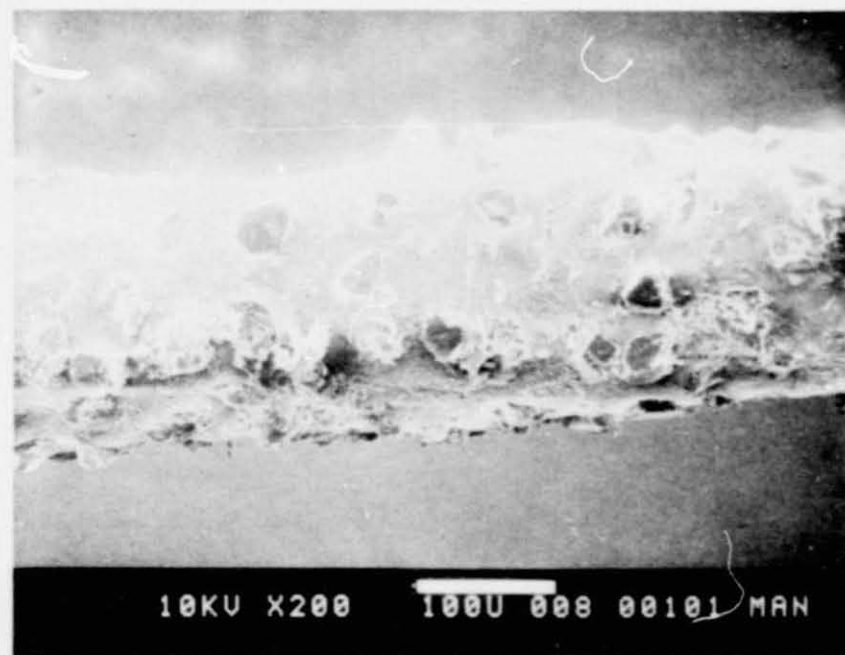


Figure 15. Three views of wire in Figure 6 after use in run 307-S.



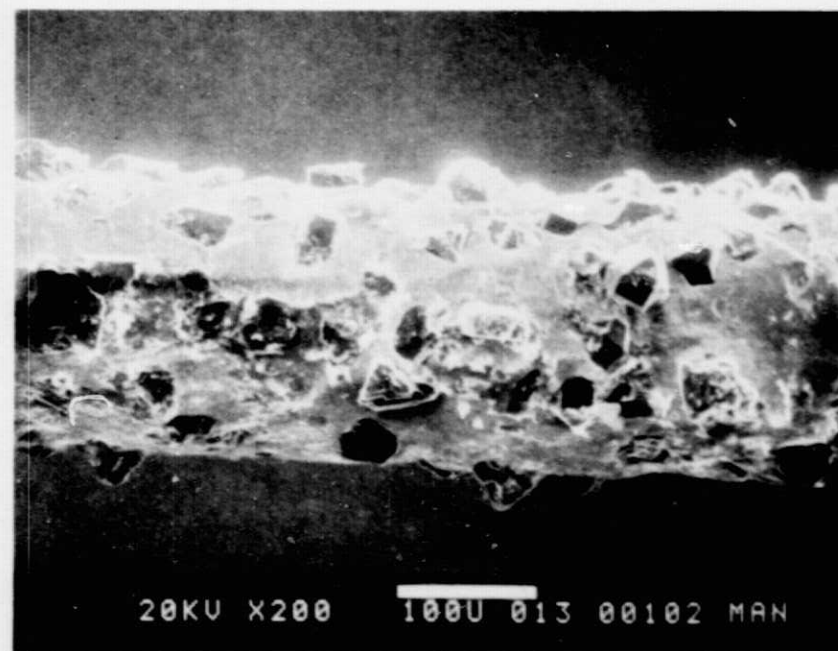
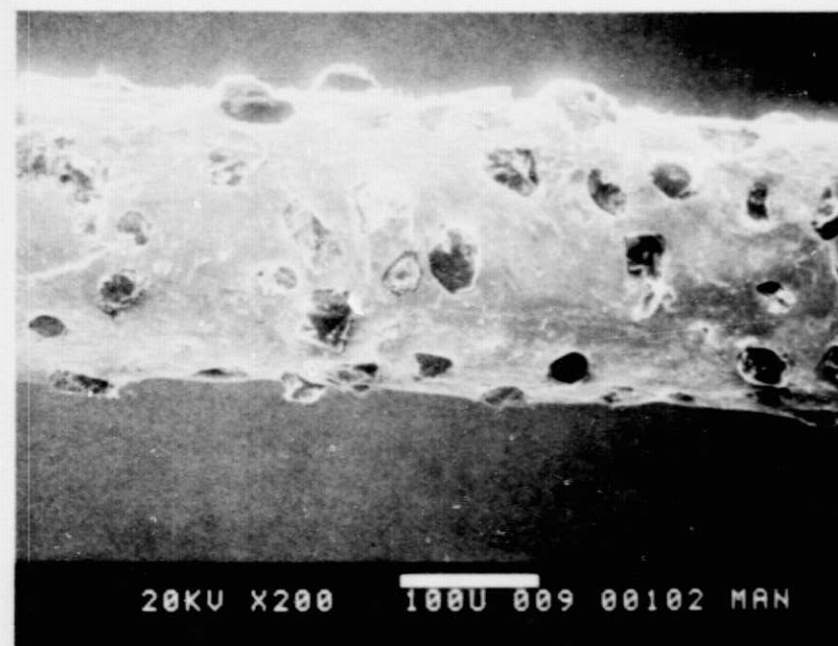
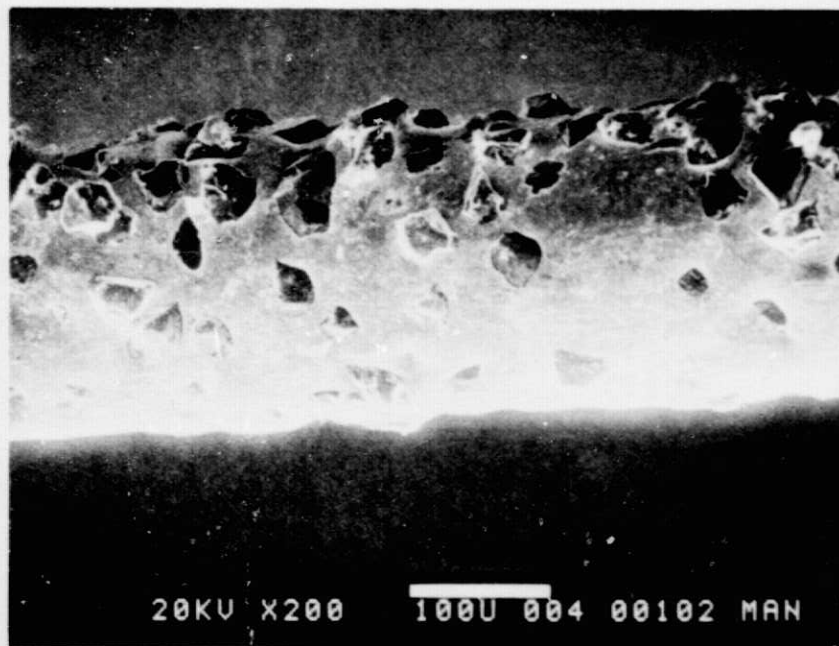


Figure 16. Three views of wire in Figure 6 after use in five runs (307-S through 311-S).

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

and an improvement of the cutting rate was achieved.

Blade development with impregnated blades has continued. A very high concentration was achieved during impregnation into 7.5 μm and 17.5 μm copper sheath. These blades were nickel plated with 7.5 μm thickness and used in runs 303-S through 306-S. In both cases it was found that good cutting performance and yields were achieved during the first slicing test; however, diamond pull-out limited the performance during the second slicing test, with the impregnation into 12.5 μm copper sheath showing a slightly better performance than the 7.5 μm sheath.

CONCLUSIONS

1. Several 20 cm diameter ingots have been cast; the largest size was 6.3 kg. The crystallinity has been good.
2. It is necessary to support large diameter crucibles, such as 20 cm, to prevent sagging and silicon spills.
3. ATJ graphite retained its dimensional integrity even on contact with molten silicon.
4. It has been experimentally demonstrated that the impurities in graphite can be removed by heating in vacuum at high temperatures.
5. In HEM the impurities are rejected to the last material to freeze. This has been confirmed by resistivity measurements.
6. The 10 cm x 10 cm cross-section crucibles are difficult to heat treat with the present setup.
7. Electroplated wires with 45 μm synthetic diamonds have been used for five runs without significant diamond pull-out.
8. Electroplated wires with 30 μm natural diamond have been used to slice four ingots without showing much degradation.
9. Kerf reduction with electroplated wires has to be accomplished with decreased nickel plating thickness and

smaller diamond size.

10. During slicing of 10 cm x 10 cm workpiece instability of the feed and rocking mechanism was observed. Modifications are in progress to remedy this effect. At the same time the bladehead will be lightened by about 50% of its weight to achieve higher speeds.

REFERENCES

1. F. Schmid and C. P. Khattak, "Heat Exchanger Method--Ingot Casting/Fixed Abrasive Method--Multi-Wire Slicing (Phase II), DOE/JPL 954373, Crystal Systems, Inc., Quarterly Progress Report No. 3, July 15, 1978.
2. F. Schmid and C. P. Khattak, "Heat Exchanger Method--Ingot Casting/Fixed Abrasive Method--Multi-Wire Slicing (II)," DOE/JPL 954373, Crystal Systems, Inc., Quarterly Progress Report No. 4, October 15, 1978.

MILESTONE CHART

